

# Efficacy of Ethiprole Applied Alone and in Combination with Conventional Insecticides for Protection of Stored Wheat and Stored Corn

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**ABSTRACT** The insecticidal pyrazole ethiprole, applied at rates of 7.5 and 10.0 ppm either alone or in combination treatments with deltamethrin, piperonyl butoxide, and chlorpyrifos-methyl, was evaluated as a protectant of stored wheat and stored corn. The commodities were treated with six treatment combinations, including an untreated control, and held for 6 mo at 22, 27, or 32°C and 57% RH. Bioassays were conducted monthly by exposing the rice weevil, *Sitophilus oryzae* (L.), and the red flour beetle, *Tribolium castaneum* (Herbst), on treated wheat and the maize weevil, *Sitophilus zeamais* (Motschulsky), and the red flour beetle on treated corn. The storage temperature of wheat did not significantly affect mortality of exposed insects ( $P \geq 0.05$ ). All rice weevils were dead after 1 wk in all treatments, and no F<sub>1</sub> adults were produced. Mortality of red flour beetles was not dependent on either chemical treatment or bioassay month, and no F<sub>1</sub> adults were produced. The storage temperature of corn did not significantly affect mortality of exposed insects ( $P \geq 0.05$ ). Mortality of maize weevils varied from 77.9 to 100% in all chemical treatments, and no F<sub>1</sub> adults were produced. Mortality of red flour beetles was also variable among treatments and bioassay month and no F<sub>1</sub> adults were produced. This is the first published report of a study in which pyrazoles have been evaluated against stored-grain insects.

**KEY WORDS** ethiprole, rice weevil, maize weevil, red flour beetle, wheat, corn

INSECTICIDAL PYRAZOLES ARE a new family of insecticides that act on the GABA (gamma-amino-butric acid) receptors of insects by blocking the passage of chloride ions, thereby causing disruption of the central nervous system (Cole et al. 1993). The mode of action of pyrazoles is similar to cyclodienes, and in general pyrazoles are highly specific to insects and are considered to have low toxicity against mammals. Fipronil, one pyrazole compound, was developed in 1987 by scientists at Aventis (formerly Rhone-Poulenc) in the United Kingdom (Mulrooney and Goli 1999). Fipronil has broad-spectrum activity against many agricultural pests, and numerous tests have demonstrated increased efficacy relative to compounds currently used in management programs (Hamon et al. 1996). Activity has been demonstrated against other insect and arthropod pests, including cockroaches (Gahlhoff et al. 1999, Silverman and Liang 1999), cat fleas (Harvey et al. 1997, Hutchinson et al. 1998), desert locusts (Rachadi and Foucart 1999), ticks (Davey et al. 1999), and thrips (Christian et al. 1997, Ester et al. 1997).

Currently many organophosphate insecticides commonly used in agricultural and urban pest management systems are being reviewed under the Environmental Protection Agency (EPA) interpretation of the 1996 Food Quality Protection Act (FQPA). The use of malathion, chlorpyrifos-methyl, and pirimiphos-methyl on grains may be restricted by those EPA reviews. Therefore, alternative treatments may be needed and pyrazoles may be potential replacement compounds. Fipronil is currently being evaluated in many agricultural markets and has the potential for numerous registrations in field crops. Fipronil may not be registered for the postharvest market because potential accumulated residues on other crops may exceed the recommended average daily intake of the compound. However, ethiprole is another pyrazole that could be considered for the postharvest market. Ethiprole is similar in toxicity, chemical structure and mode of action to fipronil, but there are no published data concerning its efficacy against stored-product insects.

Most trials with pyrazoles have related to agricultural or urban insect pests. Objectives of this study were as follows: (1) evaluate ethiprole alone or in combination with other insecticides as residual protectants of stored hard red winter wheat and stored corn, (2) determine effects of temperature on degra-

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dation of residual activity, and (3) assess impact of exposure on progeny production. Insects used in bioassays on wheat were the rice weevil, *Sitophilus oryzae* (L.), an internal feeder, and an external feeder, the red flour beetle, *Tribolium castaneum* (Herbst). Insect species used for bioassays on corn were an internal feeder, the maize weevil, *S. zeamais* (Motchulsky), and the red flour beetle.

### Materials and Methods

Treatments evaluated during the study were as follows: (1) untreated controls, (2) 10 ppm ethiprole, (3) 10 ppm ethiprole + 10 ppm piperonyl butoxide (PBO), (4) 7.5 ppm ethiprole + 0.25 ppm deltamethrin + 2 ppm PBO, (5) 10 ppm ethiprole + 0.25 ppm deltamethrin + 2 ppm PBO, and (6) 7.5 ppm ethiprole + 3 ppm chlorpyrifos-methyl. Emulsifiable concentrate (EC) formulations of all of these insecticides were obtained from Aventis (Research Triangle Park, NC), and the concentration of each formulation was as follows: ethiprole, 100 mg (AI)/ml; deltamethrin, 25 mg (AI)/ml; piperonyl butoxide, 960 mg (AI)/ml; and chlorpyrifos-methyl, 480 mg (AI)/ml. Each treatment was formulated using tap water in volumetric flasks. The untreated controls consisted of tap water alone.

Each replicate consisted of 1.5 kg hard red winter wheat, and there were four replicates for each treatment. Insecticide treatments for each replicate were applied at the rate of 1.05 ml of formulated spray per 1.5 kg of wheat to correspond to the labeled spray rate for chlorpyrifos-methyl. A Badger 100 artist's air brush (Franklin Park, IL) was used to mist each insecticidal treatment directly onto the layer of 1.5 kg wheat, which was spread out on a plywood sheet measuring 0.6 by 0.3 m. Untreated wheat was sprayed at the same rate with the tap water. After the untreated and the treated wheat were sprayed, the 1.5-kg lots were hand-rolled inside a 3.8-liter glass jar for about 30 s to ensure an even distribution. Each replicate lot was then subdivided by filling each of 42 plastic vials with  $\approx 30$  g of treated wheat. The vials were capped with a screened lid, and the remaining wheat was discarded.

Eighteen plastic boxes measuring 26 by 36.5 by 15 cm with waffle-type grids cut to fit the bottom were filled with about 750 ml of saturated NaBr to maintain an approximate relative humidity of 57–60% (Green-span 1977). Six boxes were placed in each of three temperature incubators set at 22, 27, and 32°C, one box for each treatment. Vials containing wheat for each treatment were put in one of the humidity boxes in each incubator, and each box contained a total of 56 vials (7 for each monthly bioassay at 0.5, 1, 2, 3, 4, 5, and 6 mo posttreatment  $\times$  1 set of four for each species). At each bioassay interval, two sets of four vials were removed from each of the three temperature incubators (12 vials for each species). Twenty mixed-sex 1- to 2-wk-old adult rice weevils or 20 mixed-sex 1- to 2-wk-old adult red flour beetles were put in individual vials in each set of 12. These vials were then placed inside a fourth temperature incu-

bator set at 27°C in a new plastic box containing saturated NaBr, and adult rice weevils and adult red flour beetles were exposed for 1 and 2 wk, respectively. These bioassays were conducted at 27°C because previous studies have indicated that 27°C and 57% RH was optimum for both species in terms of progeny development (Arthur 2002).

Upon completion of the exposure interval, insects were removed from the wheat and mortality was assessed for both species. Beetles were classified as dead if they did not move when prodded with a probe. The wheat was then put back into the vials, returned to the 27°C incubator, and held for 8 wk to record emergence of  $F_1$  adults. After the  $F_1$  adults were counted the wheat was discarded.

Procedures were repeated on corn using exactly the same insecticidal treatments and methods as described above, except that the individual bioassay vials contained 25 g of corn, and the maize weevil was used instead of the rice weevil. Results were analyzed separately for each commodity, with insect species, insecticide treatments, storage temperature, and residual bioassay month as main effects. Variables for analysis were initial mortality and the number of  $F_1$  adults. The analysis of variance (ANOVA), REG, and GLM procedures of the SAS (SAS Institute 1987) were used in the data analysis.

### Results

**Hard Red Winter Wheat.** Mortality (mean  $\pm$  SE) of red flour beetle and rice weevil on untreated wheat was only  $1.8 \pm 0.9$  and  $0.8 \pm 0.4\%$ , respectively, and these data were eliminated from the analysis. For the five insecticide treatments, main effects (1) species ( $F = 4,126.1$ ;  $df = 1, 630$ ), (2) insecticide treatment ( $F = 35.1$ ;  $df = 4, 630$ ), and (3) residual bioassay month ( $F = 96.4$ ;  $df = 6, 630$ ) were all highly significant ( $P < 0.01$ ). The temperature at which the wheat was stored was not significant ( $F = 118.9$ ;  $df = 2, 630$ ,  $P = 0.20$ ) with respect to mortality of exposed insects, therefore data were combined to analyze differences among treatments. Mortality of rice weevils on treated wheat was 100% for all treatments and residual bioassays, and no  $F_1$  adults were produced. The number of  $F_1$  adult rice weevils in untreated wheat stored at 22, 27, and 32 was  $60 \pm 6.2$ ,  $30.2 \pm 4.0$ , and  $41.0 \pm 7.2$ , respectively.

Data for red flour beetle mortality after 2 wk of exposure on treated wheat appear in Table 1. Although the ANOVA showed significance due to bioassay month, it was primarily because of variation in the data from month to month, and not from any trend or relationship between mortality and residual bioassay.  $R^2$  values for treatments 2 through 5 were 0.05–0.19 ( $P \geq 0.05$  for all), indicating no significant correlation between mortality and bioassay month. The  $R^2$  value for treatment 6 was 0.49 ( $P < 0.05$ ), however, mortality data were still variable.

The number of  $F_1$  adult red flour beetles on untreated wheat was not significantly affected ( $P \geq 0.05$ ) by bioassay month or by the temperature at which the

**Table 1.** Percent mortality (mean  $\pm$  SEM) of adult red flour beetles exposed for 2 wk on wheat treated with insecticides: (2) 10 ppm ethiprole, (3) 10 ppm ethiprole + 10 ppm piperonyl butoxide (PBO), (4) 7.5 ppm ethiprole + 0.25 ppm deltamethrin + 2 ppm PBO, (5) 10 ppm ethiprole + 0.25 ppm deltamethrin + 2 ppm PBO, and (6) 7.5 ppm ethiprole + 3 ppm chlorpyrifos-methyl

Treatment	Bioassay month						
	0.5	1	2	3	4	5	6
2	66.2 $\pm$ 3.8c	28.3 $\pm$ 4.5c	81.2 $\pm$ 3.6ab	63.0 $\pm$ 3.0b	67.9 $\pm$ 2.8bc	42.9 $\pm$ 3.3b	29.2 $\pm$ 1.9c
3	72.1 $\pm$ 4.5cd	34.2 $\pm$ 4.3c	86.2 $\pm$ 2.3a	60.0 $\pm$ 3.7b	60.0 $\pm$ 3.8c	42.5 $\pm$ 3.4b	29.7 $\pm$ 2.7c
4	78.7 $\pm$ 3.6b	69.2 $\pm$ 4.0ab	79.2 $\pm$ 3.6ab	75.0 $\pm$ 2.9a	82.5 $\pm$ 3.7a	61.2 $\pm$ 4.9a	48.3 $\pm$ 4.1ab
5	72.5 $\pm$ 3.7cd	64.5 $\pm$ 3.87b	73.7 $\pm$ 4.7b	78.3 $\pm$ 3.1a	80.0 $\pm$ 3.9a	62.1 $\pm$ 7.6a	53.3 $\pm$ 2.9ab
6	98.7 $\pm$ 0.7a	76.7 $\pm$ 5.6a	70.0 $\pm$ 4.6b	48.4 $\pm$ 7.5c	75.0 $\pm$ 3.2ab	34.1 $\pm$ 5.1b	36.7 $\pm$ 1.3b

Mortality for adult rice weevils exposed for 1 wk was 100% for all treatments and bioassays. Mortality of red flour beetles with respect to storage temperature was not significant ( $P \geq 0.05$ ), data combined for analysis. Wheat was stored at 22, 27, and 32°C, bioassays were conducted at 0.5–6 mo posttreatment. Means within columns followed by the same letter are not significantly different ( $P \geq 0.05$ , Waller-Duncan  $k$ -ratio  $t$ -test).

wheat was stored. An average of  $22.8 \pm 1.4$  adults were collected from vials of untreated control wheat. No  $F_1$  adult red flour beetles were produced on the treated wheat in any residual bioassay, indicating either exposed adults did not reproduce or immature stages died before reaching the adult stage.

**Corn.** Mortality (mean  $\pm$  SE) of red flour beetle and maize weevil on untreated corn was 0 and  $3.8 \pm 0.06\%$ , respectively, so we eliminated these controls from the statistical analysis. For the five insecticide treatments, main effects (1) species ( $F = 1,388.9$ ;  $df = 1, 630$ ), (2) insecticide treatment ( $F = 199.5$ ;  $df = 4, 630$ ), (3) residual bioassay month ( $F = 19.8$ ;  $df = 6, 630$ ), and (4) temperature at which the wheat was stored ( $F = 4.5$ ;  $df = 1, 630$ ) were all highly significant ( $P < 0.01$ ). The overall ANOVA indicated a difference in mortality with respect to storage temperature, however, when data for each treatment-bioassay month combination were analyzed for differences among the three temperatures, only four out of the possible 35 comparisons (7 exposure intervals  $\times$  5 treatments) for each species were significant ( $P < 0.05$ ). Also, data were variable and there was no consistent temperature effect at these four combinations, therefore data

for storage temperature were combined to analyze for differences between treatments.

Data for red flour beetle mortality after 2 wk of exposure on treated corn appear in Table 2. Although the ANOVA showed significance due to bioassay month, similar to the results for wheat, this significance was apparently related to variation in the data from month to month, and not from a decline in efficacy during storage. Regressions for mortality as the dependent variable and bioassay month as the independent variable were not significant ( $P \geq 0.05$ ) for treatments 2, 3, and 4, and even through regressions for treatments 5 and 6 were significant ( $P < 0.05$ ),  $R^2$  values were only 0.09 and 0.10, respectively. However, in contrast to data for red flour beetles exposed on wheat, mortality was consistently lower in treatment 6 compared with the other five treatment combinations.

Data for maize weevil mortality after 1 wk of exposure on the treated corn also appear in Table 2. The maize weevil appeared to be susceptible to all the treatments, and mortality ranged from 77.9 to 100% for all of the residual bioassays. Data for bioassays were also quite variable, and regressions for treatments 2

**Table 2.** Percent mortality (mean  $\pm$  SEM) of adult red flour beetles exposed for 2 wk and maize weevil exposed for 1 wk on corn treated with insecticides: (2) 10 ppm ethiprole, (3) 10 ppm ethiprole + 10 ppm piperonyl butoxide (PBO), (4) 7.5 ppm ethiprole + 0.25 ppm deltamethrin + 2 ppm PBO, (5) 10 ppm ethiprole + 0.25 ppm deltamethrin + 2 ppm PBO, and (6) 7.5 ppm ethiprole + 3 ppm chlorpyrifos-methyl

Treatment	Bioassay month						
	0.5	1	2	3	4	5	6
Red flour beetles							
2	76.7 $\pm$ 3.7b	83.7 $\pm$ 2.5ab	85.0 $\pm$ 3.6a	67.9 $\pm$ 4.1b	93.7 $\pm$ 1.7a	87.5 $\pm$ 4.2a	73.3 $\pm$ 3.8a
3	87.0 $\pm$ 1.9a	86.2 $\pm$ 3.0a	94.2 $\pm$ 1.9a	87.5 $\pm$ 2.7a	91.7 $\pm$ 1.5a	92.0 $\pm$ 1.9a	84.5 $\pm$ 3.5a
4	1.7 $\pm$ 0.7c	60.4 $\pm$ 11.2bc	52.5 $\pm$ 8.1b	26.2 $\pm$ 9.2d	39.2 $\pm$ 8.5b	46.7 $\pm$ 10.7b	70.4 $\pm$ 7.6a
5	1.7 $\pm$ 0.0c	46.2 $\pm$ 10.4c	30.0 $\pm$ 6.3c	44.2 $\pm$ 10.c	42.5 $\pm$ 8.5b	42.9 $\pm$ 9.9b	41.2 $\pm$ 9.5b
6	0.8 $\pm$ 0.8c	56.7 $\pm$ 10.2c	20.0 $\pm$ 4.7c	5.8 $\pm$ 1.8e	19.5 $\pm$ 3.2c	10.0 $\pm$ 8.8c	20.8 $\pm$ 5.1c
Maize weevil							
2	92.5 $\pm$ 1.9a	95.8 $\pm$ 1.2a	99.2 $\pm$ 0.8a	91.7 $\pm$ 2.8b	99.6 $\pm$ 0.4a	98.7 $\pm$ 0.6a	98.7 $\pm$ 0.9a
3	92.9 $\pm$ 2.1a	95.0 $\pm$ 1.1a	100 $\pm$ 0.0a	98.3 $\pm$ 0.7a	100 $\pm$ 0.0a	99.2 $\pm$ 0.8a	99.2 $\pm$ 0.6a
4	77.9 $\pm$ 1.8b	82.0 $\pm$ 2.7c	92.1 $\pm$ 2.0b	93.7 $\pm$ 0.9ab	99.5 $\pm$ 0.4a	87.5 $\pm$ 2.0b	93.3 $\pm$ 2.4b
5	78.7 $\pm$ 2.7b	77.5 $\pm$ 1.8d	96.2 $\pm$ 1.6ab	90.4 $\pm$ 1.9b	96.7 $\pm$ 1.1b	81.2 $\pm$ 3.4c	97.9 $\pm$ 1.0a
6	96.2 $\pm$ 2.0a	87.0 $\pm$ 1.5b	92.1 $\pm$ 4.1ab	92.0 $\pm$ 1.9b	96.2 $\pm$ 1.4b	90.4 $\pm$ 2.0b	97.0 $\pm$ 1.3ab

Mortality of red flour beetles and maize weevils with respect to storage temperature was not significant ( $P \geq 0.05$ ), data combined for analysis. Corn was stored at 22, 27, and 32°C, bioassays were conducted at 0.5 to 6 mo posttreatment. Means within columns followed by the same letter are not significantly different ( $P \geq 0.05$ , Waller-Duncan  $k$ -ratio  $t$ -test).

and 6 with mortality as the dependent variable and bioassay month as the independent variable were not significant ( $P \geq 0.05$ ). Regressions for treatments 3, 4, and 5 were significant ( $P < 0.05$ ), with  $R^2$  values of 0.30, 0.40, and 0.23, respectively.

The number of  $F_1$  adult red flour beetles and  $F_1$  adult maize weevils on untreated corn was not significant ( $P \geq 0.05$ ) with respect to regressions with bioassay month as the independent variable or for differences among the three temperatures at which the corn was stored. An average of  $20.4 \pm 1.0$  adult red flour beetles and an average of  $25.5 \pm 1.3$  adult maize weevils were collected from the bioassay vials that contained untreated corn. No  $F_1$  adult red flour beetles or maize weevils were produced on any of the insecticide-treated corn, again indicating that either exposed adults did not reproduce or immatures died before reaching the adult stage.

### Discussion

The results of this study show that the insecticidal pyrazole ethiprole, applied alone or in combination with other insecticides, can control adult rice weevils in stored wheat and adult maize weevils in stored corn. All treatments either killed exposed weevils before they could oviposit into the kernel, or somehow contaminated eggs during the oviposition process so that the eggs didn't hatch or the larvae died before they could reach the adult stage. Although red flour beetles survived exposure in the various treatments on both wheat and corn, no  $F_1$  adults were produced, indicating suppression and elimination of the next generation. Similar results were noted in previous research studies with pyrethroid insecticides applied alone or in combination with organophosphates. In these studies red flour beetles also survived after 1–2 wk of exposure, but no  $F_1$  adults were produced in the treated wheat (Arthur 1992, 1994a, 1994c).

To my knowledge this is the first published report of a study in which pyrazoles have been evaluated against stored-grain insects. Many of the recent published studies with fipronil have been conducted as trials for field crop pests, and application rates are given on a per acre or per hectare basis (Mulrooney et al. 1998, Mulrooney and Goli 1999, Rachadi and Foucart 1999, Stout et al. 2000). It is difficult to compare the results of these studies with this one because of the difference between the target insects and the method of application. Also, there are no published results using the insecticide ethiprole, all previous work has been with fipronil.

The labeled rate for chlorpyrifos-methyl applied to stored wheat in the United States is 6 ppm, while the labeled rate for pirimiphos-methyl applied to corn is 6–8 ppm. The amount of calculated ethiprole (AI) in the various treatments was either 7.5 or 10 ppm, depending on the specific treatment combination, and although these rates were slightly higher than the current rates for the organophosphates, they were used to ensure control of the internal feeders. Previous research with pyrethroids produced mixed results; res-

methrin and bioresmethrin application rates of at least 5 ppm were necessary to give residual control of rice weevils and maize weevils for 6 mo (Arthur 1992), whereas application rates of 1–2 ppm of deltamethrin and cyfluthrin did give residual control (Arthur 1994a, 1994b). Also, application rates of pyrethroids needed to kill the lesser grain borer, *Rhyzopertha dominica* (F.), which is also an internal beetle pest of stored grain, are generally much lower than those required to kill rice weevils or maize weevils (Bengston et al. 1987, Samson and Parker 1989; Arthur 1994a, 1994b).

The results of this study show that ethiprole applied alone or in combination with either piperonyl butoxide synergist or PBO plus deltamethrin would effectively control rice weevils in stored wheat and maize weevils in stored corn, two primary pests of these commodities, and prevent progeny development of the red flour beetle, an important secondary pest of both stored commodities. Currently no combination insecticidal treatments are registered for stored grains in the United States, though they are prevalent in other parts of the world, particularly Australia (White and Leesch 1996). Also, the exact economic costs of ethiprole applied alone or in combination with other insecticides have not yet been determined for various grain commodities.

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